

FURACA #04

[A-16]

Aim: Preparation of URACA from 7-Ac
by new route

eq. to 50 gm 7-Ac

AGE T PREPARATION OF TFA

RAW MATERIALS

2 Furanyl chloride	: 23.0 ml
Dmw	: (350 + 15) ml
NASH (25% excess)	: 37.5 g
1:1 HCl	: 46.0 ml
1 Dmw	: 175.0 ml
1 NaHCO ₃	: 20.4 g
1 EtOAc	: 100.0 ml
1:1 HCl	: 42.0 ml
1 EtOAc	: 250.0 ml

PROCEDURE

1. Charge Dmw 350 ml & NASH at RT
Flush the funnel & 15 ml Dmw
2. Stir at RT to get a clear soln
3. Add 2 Furanyl chloride in up to 45% at 20-25
mon stir for 5"
4. Charge EtOAc 250 ml and mon adjust
the pH to 1.0 - 0.9 by 1:1 HCl
at 20-25°C in 10-15'
5. Separate the layers
Give the O/L by HPLC analysis
6. To the O/L charge Dmw (175 ml)
mon adjust the pH to 0.0 - 0.2 by
NaHCO₃ at 20-25°C in 10-15'
7. Separate the layers
To the aq. layer charge EtOAc 100 ml
mon adjust the pH to 1.0 - 0.9 by
1:1 HCl at 20-25°C
8. Separate the layers

- keep the org phase for each stage

2-A

Vol. of OL3 : 132.0 ml

HPLC Analysis Report

RT: 9.94 min

OL3 : 132.0 ml

AL3 : 99.41

OL3 : 99.39

PREPARATION OF FURACA

RAW MATERIALS

2-ACA : 50.0 g

EEOAC : 200.0 ml

HOAC : 30.0 ml

BEF3 : 68.5 g

TFA : 32.0 ml

DMH : 600.0 ml

SHS : 1.0 g

20% NH₄ aq.

DMH : (50 + 150 + 50) ml } spray

EEOAC : (50 + 150 + 50) ml } spray

PROCEDURE

1. Charge 2-ACA into the mix of

BEF3 purged EEOAC + HOAC at 15°C

2. Add the SHS for 5 l at 15°C

then add TFA soln. then raise

the temp to 30°C

3. Maintain the temp at 30°C

up to complete rxn.

4. After completion of rxn. transfer

the mass into the DMH (600 ml)

at 15°C then add SHS.

5 Adjust to DN 10 - 2.5 3hr 18 - 20%

Nett 20in of 20 - 25 C 2 in 25 - 30

6 shift for 201 1st 20 25 C

7 Filter 11 2 2000 5 DMW & FIDAC

DURABOND PRO Monitoring Results

7 ACA 2 TFA EURACA imp

45 7.92 19.43 20.51

75

1hr 15 2.55 113.67 21.49

1hr 45 1.10 11.92 26.44

2hr 15 0.45 11.09 18.52

Dry wt = 43.72g m/c - 2

Assay 93.9% (0.03)